

PCTWORLD INTELLECTUAL PROPERTY ORGANIZATION
International Bureau

INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification ⁵: C11D 17/06, 1/72	A1	(11) International Publication Number: WO 94/16052 (43) International Publication Date: 21 July 1994 (21.07.94)
(21) International Application Number: PCT/EP93/03624 (22) International Filing Date: 17 December 1993 (17.12.93) (30) Priority Data: 9300311.9 8 January 1993 (08.01.93) GB (71) Applicant (for AU BB CA GB IE LK MN MW NZ SD only): UNILEVER PLC [GB/GB]; Unilever House, Blackfriars, London EC4P 4BQ (GB). (71) Applicant (for all designated States except AU BB CA GB IE LK MN MW NZ SD): UNILEVER N.V. [NL/NL]; Weena 455, NL-3013 AL Rotterdam (NL). (72) Inventors: HOUGHTON, Mark, Phillip; Berliozplein 1, NL- 2651 VG Berkel en Rodenrijs (NL). GORTEMAKER, Fran- siscus, Hermannus; Aveling 6, NL-3271 VA Mijnsheerenland (NL). (74) Agent: GEARY, Stephen; Unilever plc, Patent Division, Colworth House, Sharnbrook, Bedford MK44 1LQ (GB).	(81) Designated States: AT, AU, BB, BG, BR, BY, CA, CH, CZ, DE, DK, ES, FI, GB, HU, JP, KP, KR, KZ, LK, LU, LV, MG, MN, MW, NL, NO, NZ, PL, PT, RO, RU, SD, SE, SK, UA, UZ, VN, European patent (AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG). Published <i>With international search report.</i>	
(54) Title: DETERGENT COMPOSITION AND PROCESS FOR PRODUCING IT		
(57) Abstract A particulate high bulk density detergent composition is provided having improved dissolution characteristics and comprising a surfactant system including one or more anionic and/or nonionic surfactants, at least one detergency builder and a dissolution aid. The dissolution aid is present in an amount from 0.01 to 2 % by weight as calculated on the composition and comprises a nonionic surfactant which is an alkoxylated aliphatic alcohol containing at least 25 alkylene oxide groups.		

FOR THE PURPOSES OF INFORMATION ONLY

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

AT	Austria	GB	United Kingdom	MR	Mauritania
AU	Australia	GE	Georgia	MW	Malawi
BB	Barbados	GN	Guinea	NE	Niger
BE	Belgium	GR	Greece	NL	Netherlands
BF	Burkina Faso	HU	Hungary	NO	Norway
BG	Bulgaria	IE	Ireland	NZ	New Zealand
BJ	Benin	IT	Italy	PL	Poland
BR	Brazil	JP	Japan	PT	Portugal
BY	Belarus	KE	Kenya	RO	Romania
CA	Canada	KG	Kyrgyzstan	RU	Russian Federation
CF	Central African Republic	KP	Democratic People's Republic of Korea	SD	Sudan
CG	Congo	KR	Republic of Korea	SE	Sweden
CH	Switzerland	KZ	Kazakhstan	SI	Slovenia
CI	Côte d'Ivoire	LI	Liechtenstein	SK	Slovakia
CM	Cameroon	LK	Sri Lanka	SN	Senegal
CN	China	LU	Luxembourg	TD	Chad
CS	Czechoslovakia	LV	Latvia	TG	Togo
CZ	Czech Republic	MC	Monaco	TJ	Tajikistan
DE	Germany	MD	Republic of Moldova	TT	Trinidad and Tobago
DK	Denmark	MG	Madagascar	UA	Ukraine
ES	Spain	ML	Mali	US	United States of America
FI	Finland	MN	Mongolia	UZ	Uzbekistan
FR	France			VN	Viet Nam
GA	Gabon				

- 1 -

DETERGENT COMPOSITION AND PROCESS FOR PRODUCING IT

The present invention relates to a detergent composition suitable for use in fabric washing applications. The invention is of especial applicability to particulate detergent compositions, containing no, or low levels of, phosphate builder and to compositions of high bulk density. The invention also relates to a process for producing this composition.

In recent years there has been a trend to reduce or eliminate phosphate builders in particulate detergent compositions. The replacement of sodium tripolyphosphate as a builder in detergent powders by a crystalline aluminosilicate (zeolite), has led to a number of difficulties with the structure and physical properties of powders. One such problem that has been encountered, is the tendency of zeolite-built powders to dispense less well in automatic washing machines than do their phosphate-built counter-parts; a higher proportion of the powder dosed into the washing machine is left in the dispenser, in the washing process leading to product wastage and clogging. The problem is especially marked at low water inlet temperatures.

The tendency towards poor dispensing has been exacerbated by the recent trend in the detergent industry towards higher bulk density powders. Detergent powders of high bulk densities from 600 to 1100 kg/m³, preferably from 700 to 1100 kg/m³, are attractive to the customer. Because the capillary diameter of the high bulk density powder is smaller than in low bulk density powders, the water penetration into the particle is slower. As a consequence, when the powder is wetted by water flowing through the dispenser the detergent particles may stick together resulting in considerable residues of wetted and adhering powder left behind in the

- 2 -

drawer. Similar problems may be encountered when applying a detergent dosing device as described in EP-A-253,419, for in-drum dosing of high density detergent powder.

5 Detergent powders typically comprise anionic and/or nonionic surfactants. Nonionic surfactants are particularly effective in removing hydrophobic soils such as hydrocarbon oils, complex fats and other long-chain unsaturated and saturated glycerides. However, when detergent powders containing
10 nonionic surfactants come into contact with aqueous solutions, the nonionic surfactant may form a viscous phase which may impede dissolution. Nonionic surfactants having a low degree of ethoxylation, generally employed because of their oily soil detergency, are especially problematic in
15 this respect.

It has now surprisingly been found that the problem of poor dissolution and/or dispersion in the wash liquor of such a powder can be overcome by incorporating in the powder a
20 relatively small amount of an alkoxylated aliphatic alcohol containing at least 25 alkylene oxide groups.

Accordingly, the present invention provides a particulate detergent composition having a bulk density of at least 600
25 g/l comprising a surfactant system, at least one detergency builder and a dissolution aid wherein the dissolution aid is present in an amount of 0.01 to 2% by weight as calculated on the composition and comprises a nonionic material which is an alkoxylated, aliphatic alcohol containing at least 25
30 alkylene oxide groups.

The invention also provides a process for the production of a particulate detergent composition having a bulk density of at least 600 g/l comprising a surfactant system, at least one
35 detergency builder and a dissolution aid wherein the

- 3 -

dissolution aid is present in an amount of from 0,01 to 2% by weight as calculated on the composition and comprises a nonionic material which is an alkoxyated aliphatic alcohol containing at least 25 alkylene oxide groups, which process comprises mixing the dissolution aid with the surfactant system and builder sequentially or simultaneously.

The invention further provides the use of an alkoxyated aliphatic alcohol containing at least 25 alkylene oxide groups, as a dissolution aid in a particulate detergent composition having a bulk density of at least 600 g/l.

Suitably a granular detergent composition according to the invention has a bulk density from 600 to 1100 kg/m³, preferably from 700 to 1100 kg/m³. Various postdosed ingredients, such as sodium carbonate, bleach material and foam depressing agent, may be added to the composition as desired. The composition may be prepared by spray-drying optionally followed by mixing or by dry-mixing/agglomeration. For obtaining high bulk densities, the composition is preferably prepared by dry-mixing/agglomeration.

As essential ingredients, the composition contains surfactant system, builder material and a dissolution aid. It is preferred that the dissolution aid is present in the composition of the invention as a separate granular component. Preferably the dissolution aid is substantially pure alkoxyated aliphatic alcohol containing at least 25 alkylene oxide groups.

The detergent compositions of the invention are characterised by a dissolution aid comprising a nonionic material which is an alkoxyated aliphatic alcohol containing at least 25 alkylene oxide groups, preferably at least 50 alkylene oxide groups, more preferably at least 80 alkylene oxide groups.

- 4 -

Preferably at least 50%, more preferably at least 80% and especially substantially all of the alkylene oxide groups are ethylene oxide. The dissolution aid is suitably present at a level of from 0.01 to 2% by weight, preferably from 0.05 to 0.5% by weight based on the composition.

The surfactant system present in the detergent composition of the invention suitably contains alkoxyated nonionic surfactants having an average degree of alkoxylation of at most 11. Suitable nonionic surfactants include condensation products of ethylene oxide with an aliphatic alcohol having from 8 to 15 carbon atoms and an average degree of ethoxylation from 2 to 10.

A preferred surfactant system comprises a mixture of two C₈₋₁₅ nonionic surfactants having an average degree of ethoxylation respectively of 2 to 5, preferably 2.5 to 4 and 6.5 to 10, preferably 6.5 to 8 which, suitably, are present in a weight ratio of 1 to 25:5. In order to obtain particularly beneficial dissolution properties it is especially preferred that the proportion of nonionic surfactant having a branched aliphatic alcohol is in the range from 10 to 60% by weight for example about 55% by weight based on the total amount of nonionic surfactant in the composition.

Nonionic detergent-active compounds and the dissolution aid together are suitably present in the compositions of the invention in a total amount of from 2 to 50% by weight, preferably from 5 to 30% by weight.

In addition to the nonionic surfactants mentioned above, other detergent-active materials may be present in the compositions of the invention. These additional detergent-active materials may be anionic (soap or non-soap), cationic,

- 5 -

zwitterionic, amphoteric surfactants, or any combination of these surfactants.

5 Anionic detergent-active compounds may be present in an amount of from 0 to 40% by weight, preferably from 0 to 20% by weight. It is preferred that the ratio of nonionic surfactant and dissolution aid to anionic surfactant is within the range of 1:2 to 9:1.

10 Synthetic anionic surfactants are well known to those skilled in the art. Examples include alkylbenzene sulphonates, particularly sodium linear alkylbenzene sulphonates having an alkyl chain length of C₈-C₁₅; primary and secondary alkyl
15 sulphates, particularly sodium C₁₂-C₁₅ primary alkyl sulphates, olefin sulphonates; alkane sulphonates; dialkyl sulphosuccinate; and fatty acid ester sulphonates.

It may also be desirable to include one or more soaps of fatty acids. These are preferably sodium soaps derived from
20 naturally occurring fatty acids, for example the fatty acids from coconut oil, beef tallow, sunflower or hardened rapeseed oil.

The total amount of surfactant in the compositions of the
25 invention is suitably from 5 to 50% by weight. Of particular interest are high-performance compositions containing relatively high levels of surfactant, preferably from 10 to 50% by weight and more preferably from 15 to 50% by weight.

30 Especially preferred compositions according to the invention include surfactant systems consisting especially of nonionic surfactant as described above in combination with linear alkylbenzene sulphonate (LAS) or primary alcohol sulphate (PAS) or both.

35

- 6 -

Surfactant systems of especial interest consist essentially of

(i) from 40 to 100% by weight of ethoxylated nonionic surfactant, and

5 (ii) from 0 to 60% by weight of linear alkylbenzene sulphonate or primary C₈-C₁₈ alcohol sulphate.

The detergent powders of the invention contain one or more detergency builders, suitably in an amount from 5 to 80% by weight, preferably from 20 to 60% by weight. The invention is especially applicable to compositions containing alkali metal aluminosilicates as builders. Alkali metal (preferably sodium) aluminosilicates may generally be incorporated in an amount from 5 to 60% by weight (anhydrous basis) of the composition, preferably from 25 to 55% by weight, and suitably, in a heavy duty detergent composition, from 25 to 46% by weight.

20 The alkali metal aluminosilicate may be either crystalline or amorphous or mixtures thereof, having the general formula:



25 These materials contain some bound water and are required to have a calcium ion exchange capacity of at least 50 mg CaO/g. The preferred sodium aluminosilicates contain 1.5-3.5 SiO₂ units (in the formula above).

30 Suitable crystalline sodium aluminosilicates ion-exchange detergency builders are described, for example, in GB 1,429,143 (Procter & Gamble). The preferred sodium aluminosilicates of this type are the well-known commercially available zeolite A and X, and mixtures thereof.

- 7 -

The zeolite may be the commercially available zeolite 4A now widely used in laundry detergent powders. Alternatively, the zeolite builder incorporated in the compositions of the invention is maximum aluminium zeolite P (zeolite MAP) as described and claimed in EP-A-384,070 (Unilever). Zeolite MAP is defined as an alkali metal aluminosilicate of the zeolite P type having a silicon to aluminium ratio not exceeding 1.33, preferably within the range of from 0.90 to 1.33, and more preferably within the range of from 0.90 to 1.20.

The calcium binding capacity of zeolite MAP is generally at least 150 mg Cao per g of anhydrous material.

Other builders may also be included in the detergent compositions of the invention if necessary or desired.

Inorganic builders that may be present include sodium carbonate, if desired in combination with a crystallisation seed for calcium carbonate, as disclosed in GB 1,437,950 (Unilever).

Organic builders that may be present include polycarboxylate polymers such as polyacrylates, acrylic/maleic copolymers, and acrylic phosphonates; monomeric polycarboxylates such as citrates, gluconates, oxydisuccinates, glycerol mono-, di- and trisuccinates, carboxymethyloxysuccinates, hydroxy-ethyliminodiacetates, alkyl- and alkenyl-malonates and succinates; and sulphonated fatty acid salts. This list is not intended to be exhaustive.

Builders, both inorganic and organic, are preferably present in alkali metal salt, especially sodium salt, form.

Especially preferred supplementary builders are polycarboxylate polymers, more especially polyacrylates and acrylic/maleic copolymers, suitably used in amounts of from 0.5 to 15% by weight, especially from 1 to 10% by weight; and

- 8 -

monomeric polycarboxylates, more especially citric acid and its salts, suitably used in amounts of from 3 to 20% by weight, more preferably from 5 to 15% by weight.

5 The compositions according to the invention may contain alkali metal, preferably sodium, carbonate, to increase detergency and to ease processing. Sodium carbonate may generally be present in amounts ranging from 1 to 60% by weight, preferably from 2 to 40% by weight, and most
10 preferably from 2 to 13% by weight. However, compositions free of alkali metal carbonate are also within the scope of the invention.

Preferred compositions of the invention preferably do not
15 contain more than 5% by weight of inorganic phosphate builders, and are desirably substantially free of phosphate builders.

Fully formulated laundry detergent compositions according to
20 the present invention may additionally contain any suitable ingredients normally employed in detergent compositions, for example, inorganic salts such as sodium silicate or sodium sulphate; organic salts such as sodium citrate; anti-redeposition aids such as cellulose derivatives and acrylate
25 or acrylate/maleate polymers; fluorescers; bleaches, bleach precursors and bleach stabilizers; proteolytic and lipolytic enzymes; dyes; coloured speckles; perfumes; foam controllers; fabric softening compounds.

30 The particulate detergent compositions of the invention may in principle be prepared by any of the available tower (spray-drying), non-tower (granulation) or combination processes.

- 9 -

Of especial interest are compositions of high bulk density - at least 600 g/l, preferably at least 700 g/l and most preferably at least 800 g/l - which may be prepared by processes involving granulation and/or densification in a high-speed mixer/granulator.

One suitable method comprises spray-drying a slurry of compatible heat-insensitive ingredients, including the zeolite MAP, any other builders, and at least part of the detergent-active compounds; densifying the resulting base powder in a mixer/granulator; and then spraying on or post-dosing those ingredients unsuitable for processing via the slurry (for example bleaches and enzymes).

In another method, the spray-drying step can be omitted altogether, a high bulk density base powder being prepared directly from its constituent raw materials, by mixing and granulating in a high-speed mixer/granulator, and then post-dosing bleach and other ingredients as in the spray-drying/post-tower densification route.

The high-speed mixer/granulator, also known as a high-speed mixer/densifier, may be a batch machine such as the Fukae (Trade Mark) FS, or a continuous machine such as the Lodige (Trade Mark) Recycler CB30.

Processes using high-speed mixer/granulators are disclosed, for example, in EP-A-340,013, EP-A-367,339, EP-A-390,251 and EP-A-420,317 (Unilever).

The dissolution aid may be included in the base powder but is preferably admixed with the finished base powder. Nonionic surfactants having a lower degree of alkoxylation, particularly those containing on average less than 11 ethylene oxide groups, may be included in the base powder, post added, or both.

- 10 -

As mentioned above, detergent compositions according to the present invention have been found to show improved dissolution properties when employed in a washing process in a washing machine. Additional benefits associated with the invention were found to be reduced redeposition characteristics during the washing cycle and improved bleeding behaviour upon storage.

EXAMPLES

The following non-limiting Examples illustrate the invention. Examples identified by numbers are in accordance with the invention, those identified by letter are comparative. Parts and percentages are by weight unless otherwise stated.

Examples 1-9, Comparative Example A

A particulate detergent composition having a bulk density of 830 kg/m³ was prepared by spray-drying an aqueous slurry to form a base powder (including nonionic surfactants as specified), densifying the base powder in a continuous Lodige high-speed mixer/granulator, spraying-on further nonionic surfactants as specified, and then admixing the remaining ingredients.

The general formulation, in weight percent, is shown in Table 1.

- 11 -

TABLE 1

	Base powder	
	Linear alkylbenzene sulphonate	8.60
5	*Nonionic surfactant (i)	1.92
	Zeolite 4A ¹	23.61
	Acrylic/maleic copolymer ²	3.51
	Sodium carbonate	7.49
	Minor ingredients ³	1.35
10	Moisture	9.02
		55.50

	Sprayed-on	
15	**Nonionic surfactant (ii)	4.68

- 12 -

	Admixed	
	Zeolite 4A	5.00
	Sodium Carbonate	4.30
5	Granular sodium silicate	3.75
	TAED ⁴	7.75
	Sodium perborate monohydrate	15.00
	EDTMP ⁵	0.37
	Antifoam granules ⁶	2.00
10	Enzyme granules	1.00
	Perfume	0.65
		100.00

15 wherein:

- * nonionic ethoxylated alcohol, Synperonic A7 (containing 7 EO groups), ex ICI
- 20 ** nonionic ethoxylated alcohol, 1:1 mixture of Synperionic A3 and A7 (containing 3 and 7 EO groups), ex ICI

1: Wessalith (Trade Mark) P powder ex Degussa: percentage is for anhydrous material, the water of hydration being

25 included in the figure for total moisture.

2: Sokalan (Trade Mark) CP5, ex BASF.

3: Fluorescer, sodium carboxymethylcellulose, salts, etc.

30

- 13 -

4: Tetraacetylenediamine, as 83%wt granules.

5: Ethylenediaminetetramethylenephosphonic acid, calcium salt: Dequest (Trade Mark) 2047 ex Monsanto (34%wt active).

5

6: Antifoam granules in accordance with EP-B-266,863 (Unilever).

10

To the thus prepared particulate detergent composition varying -minor- amounts of several types of ethoxylated alcohol containing at least 25 ethylene oxide (EO) groups, were postdosed, as specified in Table 2. Suitable commercially available nonionic materials containing at least 25 EO groups include the LUTENSOL AT [Trade Mark] series ex BASF and the BRIJ [Trade Mark] series ex ICI.

15

As can be seen, the type of the postdosed nonionic materials is indicated in this Table by number of EO-groups present therein.

20

- 14 -

TABLE 2

Example	Postdosed high EO	nonionic material
	amount (parts)	type
A	0.000	--
1	0.50	25 EO
2	0.125	25 EO
3	0.50	50 EO
4	0.125	50 EO
5	1.00	80 EO
6	0.50	80 EO
7	0.25	80 EO
8	0.125	80 EO
9	0.0625	80 EO

The delivery characteristics of the thus obtained powders were tested using a model system which simulates the delivery of a powder in an automatic washing machine.

For this test a cylindrical vessel having a diameter of 4 cm and a height of 7 cm, made of 600 micron pore size stainless steel mesh, and having a top closure made of teflon and a bottom closure made of the above type of mesh, was used. In this top closure, a 30 cm metal rod was inserted to act as a handle, and this handle was attached in an agitator arm positioned above 1 litre water present in a container and having a temperature of 20 C. By means of this agitator apparatus the cylindrical vessel held at 45 degrees, could be rotated through a circle with a 10 cm radius during 2

- 15 -

seconds. Subsequently the vessel could be allowed to rest during 2 seconds before the next rotation-rest cycle started.

5 A 50 grams powder sample was introduced in the cylindrical vessel. This vessel was then closed and attached to the agitator arm which was subsequently moved down to a position wherein the top of the cylindrical vessel was just below the water surface. After a ten second delay, the rotation was started, and the apparatus then allowed to operate for 15 rotation-rest cycles.

10 Subsequently, the cylindrical vessel and handle were removed from the water and the vessel was detached. Surface water was carefully poured off, and any powder residues transferred to a preweighed container. The container was then dried at 15 100°C for 24 hours, and the weight of dried residue as a percentage of the initial powder weight calculated.

The results are shown in Table 3.

20

TABLE 3

Example	Postdosed high EO nonionic material		Residue
	amount (parts)	type	(wt%)
A	0.000	--	34
1	0.50	25 EO	22
2	0.125	25 EO	20
3	0.50	50 EO	24
4	0.125	50 EO	18
5	1.00	80 EO	25
6	0.50	80 EO	22
7	0.25	80 EO	16
8	0.125	80 EO	14
9	0.0625	80 EO	21

It can be seen from Table 3 that the largest reduction of the powder residue as compared to the residue found in example A, could be obtained when applying a detergent powder composition comprising a minor amount of postdosed ethoxylated alcohol containing 80 ethylene oxide groups.

Examples 10,11, Comparative Example B

A particulate detergent composition was prepared in a very similar way and having almost the same formulation as in the above-mentioned Example A, the only difference being that in the present Examples the sprayed-on nonionic surfactant material fully consists of Synperionic A3 (containing 3 EO groups) ex ICI.

- 17 -

To this detergent powder, varying amounts of ethoxylated alcohol containing 80 EO groups were postdosed.

The delivery characteristics of the thus obtained powders were tested using the above-described model system and ..

5 testing process. The results obtained are shown in Table 4:

TABLE 4

Example	Postdosed high EO nonionic material		Residue
	amount (parts)	type	(wt%)
B	0.000	--	68
10	0.50	80 EO	55
11	0.125	80 EO	42

10 It can be seen that the dissolution properties of the detergent powder of Example B are inferior as compared to the powder of Example A, and that also in this case an
15 improvement of the delivery characteristics could be obtained by post-dosing minor quantities of ethoxylated alcohol containing 80 EO groups.

20 Comparative Examples C,D

To the detergent powder of Example A, varying -minor- amounts of polyethylene glycol having a molecular weight of 4000 (PEG 4000) were postdosed.

30 The delivery characteristics of the thus obtained powders were tested using the above-described model system and testing process. The results obtained are shown in Table 5.

- 18 -

TABLE 5

Example	Postdosed PEG 4000	Residue
	amount (parts)	(wt%)
A	0.000	34
B	0.50	33
C	0.25	35

It can be seen that no improvement of the delivery characteristics of the detergent powder of Example A could be obtained by post-dosing the above-indicated minor amounts of PEG 4000.

Example 12 and Comparative Example E

A detergent composition having a bulk density of about 900 g/l was prepared by a mixing/granulation process to produce a base powder to which further components were post-dosed into the base powder as listed below.

- 19 -

	Base Powder	
	Sodium primary alkyl sulphate (PAS)	5.81
	Zeolite MAP	36.04
5	Sodium carbonate	0.96
	*Synperonic A3 ex ICI	7.15
	*Synperonic A7 ex ICI	5.81
	Stearic acid	2.04
	bTallow 80EO	0.20
10	Sodium carboxymethyl cellulose	0.89
	Moisture	4.98
		63.88

a About 55% branched.

b Ethoxylated tallow alcohol having an average degree of ethoxylation of 80.

The nonionic materials (*) were mixed and then sprayed onto an adjunct comprising PAS, carbonate and part of the zeolite, and sodium carboxymethyl cellulose and stearic acid in a Lodige CB30 "Recycler" mixer. The stearic acid was neutralised by addition of base. The mixture was then layered with the remaining zeolite and passed to a Lodige KM300 "Ploughshare" mixer and then a fluid bed as described in EP-A-367 339 (Unilever) to produce the base powder.

The following components were then admixed to the base powder (63.88 parts).

- 20 -

	Fluorescer	3.00
	Sodium silicate	2.90
	^c TAED	4.75
	^d Manganete catalyst	2.40
5	Sodium Percarbonate	20.50
	^e DEQUEST 2047 ex Monsanto	0.37
	Enzyme	1.75
	Perfume	0.45

- 10 ^c tetraacetyl ethylene diamine
 ^d as described in EP 458 397 (Unilever)
 ^e sequestrant

15 A comparative composition E was prepared by the same process
 and to the same composition as Example 12 save that the
 Tallow 80EO in the base was replaced by 0.20 parts SYNPERONIC
 A3.

20 The delivery characteristics of the two powders were tested
 using the model system described in Examples 1 to 9. The
 results are shown in Table 6.

TABLE 6

25	Composition	Residue (wt%)
	12	45
	E	60

30 These results demonstrate that compositions containing a
 dissolution aid have significantly superior delivery

- 21 -

characteristics when compared to a similar composition which does not contain a dissolution aid.

Detergency

The detergencies of the powders of Examples A,D (comparative) and 7 (according to the invention) were compared by means of a washing machine test. The machine used was a Siemens Siwamat (Trade Mark) Plus 3700 front-loading automatic washer.

3 kg soiled loads containing white cotton interlock test cloth monitors (which monitors have been pre-washed) were washed at 40°C using the half load main wash programme. The powder samples (104 g dose) predissolved in 250 ml water were introduced via the machine's dispenser. Detergency results (reflectance loss after 10 washes at 460 nm of the test cloth monitors) were as follows:

Composition of	Reflectance loss after 10 washes
Example A	2.1
Example D	1.9
Example 7	1.4

These results demonstrate the additional benefit associated with the invention, of reduced redeposition.

Bleeding

- 22 -

The bleeding characteristics of the powders of Examples A,D (comparative) and 7 (according to the invention) were compared. To that end, Standard VC2 cardboard packs (having a volume of 1.5 litre) were filled up to 80% of their total volume with samples of the above mentioned respective powders. Subsequently, the filled cardboard packs were stored at a temperature of 37°C and a relative humidity of 70%, for two months. The part of the total cardboard interior surface which was stained and had obtained a darker colour due to nonionic migration (bleeding) during this two months storage period, was recorded as a percentage, as is shown below:

Composition of	stained part of cardboard pack interior surface (in %)
Example A	18
Example D	19
Example 7	8

It can be derived from these results that a further benefit associated with the invention, is significantly improved (i.e. reduced) bleeding behaviour of detergent compositions according to the invention.

CLAIMS

1. A particulate detergent composition having a bulk density of at least 600 g/l and comprising a surfactant system including one or more anionic and/or nonionic surfactants, at least one detergency builder and a dissolution aid, wherein the dissolution aid is present in an amount from 0.01 to 2% by weight as calculated on the composition and comprises a nonionic material which is an alkoxyated aliphatic alcohol containing at least 25 alkylene oxide groups.
2. A composition according to claim 1 in which at least 50% of the alkylene oxide groups in the nonionic material are ethylene oxide groups.
3. A detergent composition according to claim 1 or claim 2, comprising from 0.05 to 1% by weight of the dissolution aid.
4. A detergent composition according to any preceding claim, wherein the alkoxyated aliphatic alcohol contains at least 50 ethylene oxide groups.
5. A detergent composition according to any preceding claim, wherein the surfactant system comprises two nonionic surfactants which are condensation products of ethylene oxide with a C₈-C₁₅ aliphatic alcohol having respectively an average degree of ethoxylation of 2 to 5 and of 6.5 to 10.
6. A detergent composition according to any preceding claim, wherein the surfactant system consists essentially of
- (i) from 40 to 100% by weight of the surfactant system of ethoxyated nonionic surfactant, and
 - (ii) from 0 to 60% by weight of linear alkylbenzene sulphonate or primary C₈-C₁₂ alcohol sulphate.

- 24 -

7. A detergent composition according to any preceding claim in the surfactant system is present in an amount from 10 to 50% by weight of the composition.

5 8. A detergent composition according to any preceding claim, which is essentially free of phosphates.

9. A process for the production of a particulate detergent composition having a bulk density of at least 600 g/l comprising a surfactant system, at least one detergency builder, and a dissolution aid wherein the dissolution aid is present in an amount of from 0.01 to 2% by weight as calculated on the composition and comprises a nonionic material which is an alkoxyated aliphatic alcohol containing at least 25 alkylene oxide groups, which process comprises mixing the dissolution aid with the surfactant system and builder sequentially or simultaneously.

10. Use of an alkoxyated aliphatic alcohol containing at least 25 alkylene oxide groups, as a dissolution aid in a particulate detergent composition having a bulk density of at least 600 g/l.

INTERNATIONAL SEARCH REP RT

International application No. . . .

PCT/EP 93/03624

A. CLASSIFICATION OF SUBJECT MATTER

IPC5: C11D 17/06, C11D 1/72

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC5: C11D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

WPI

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US, A, 4137197 (JOHN KOWALCHUK), 30 January 1979 (30.01.79), abstract; column 1, line 5-20; column 4, line 1-10; claims 1, 16, 24-25	1,2,7,8
P,X	WO, A1, 9302176 (HENKEL KOMMANDITGESELLSCHAFT AUF AKTIEN), 4 February 1993 (04.02.93), abstract; page 4, line 30 - page 5, line 1-12; claims 1, 3	1-10
P,X	EP, A1, 0561103 (THE PROCTER & GAMBLE COMPANY), 22 Sept 1993 (22.09.93), page 2, line 3-5; page 3 - page 4, line 1-18; claims 1-2	1-10

☒ Further documents are listed in the continuation of Box C.☒ See patent family annex.

* Special categories of cited documents

- *A* document defining the general state of the art which is not considered to be of particular relevance
- *E* earlier document but published on or after the international filing date
- *L* document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
- *O* document referring to an oral disclosure, use, exhibition or other means
- *P* document published prior to the international filing date but later than the priority date claimed

T later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

X document of particular relevance: the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

Y document of particular relevance: the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

& document member of the same patent family


Date of the actual completion of the international search

11 April 1994

Date of mailing of the international search report

29. 04. 94

Name and mailing address of the ISA/



European Patent Office, P.O. Box 3818 Patenzan 2
NL-2280 HV Rijswijk
Tel. (+31-70) 340-2040, Tx. 31 651 epo nl,
Fax: (+31-70) 340-3016

Authorized officer

DAGMAR JÄRVMAN

INTERNATIONAL SEARCH REPORT

International application No.

PCT/EP 93/03624

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
P, A	<p>EP, A1, 0578871 (THE PROCTER & GAMBLE COMPANY), 19 January 1994 (19.01.94)</p> <p style="text-align: center;">-- -----</p>	1-10

S/ 3668

INTERNATIONAL SEARCH REPORT
Information on patent family members

26/02/94

International application No.

PCT/EP 93/03624

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US-A- 4137197	30/01/79	CA-A- 1112122	10/11/81
WO-A1- 9302176	04/02/93	DE-A- 4124701	28/01/93
EP-A1- 0561103	22/09/93	AU-A- 3919493	21/10/93
		WO-A- 9319148	30/09/93
EP-A1- 0578871	19/01/94	NONE	

10

10

10

10

10

10

10

10

10

10

10

10

10

10

10

10

10

10

10

10

10

10

10

10

10

10

10

10

10

10

10

10

10

10

10

10